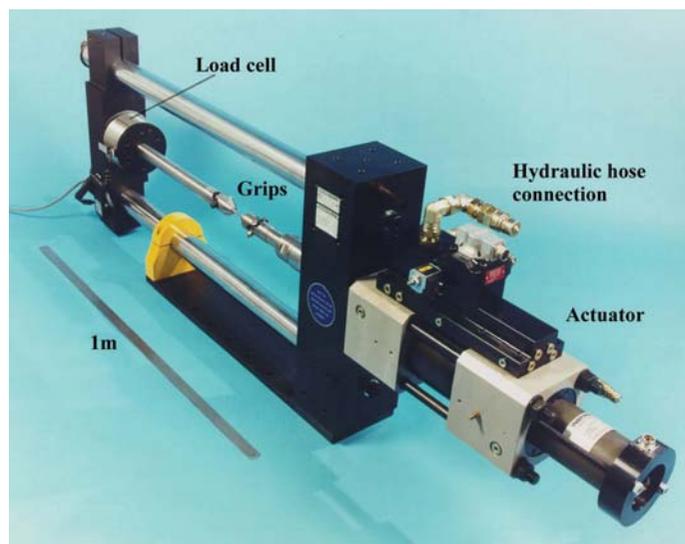


## 2. INSTRUMENTATION AND SAMPLE PREPARATION

**Figure 2.3.27**

Elements of a typical mechanical testing machine used for applying uniaxial stress (pressure) to samples on an engineering neutron diffractometer. This example of a 100 kN device is from the instrument ENGIN-X at the ISIS facility, UK. (Credit: STFC.)

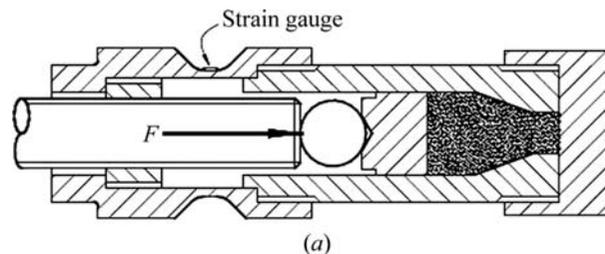
Non-ambient temperature devices are usually designed for operation either below or above ambient temperature. However, there are a large number of phase transitions and other phenomena that span from below to above ambient temperature. In order to avoid transferring samples from one sample environment to another mid-experiment, a useful hybrid device is the cryo-furnace. Cryo-furnaces are based around the liquid-helium cryostat and are equipped with more powerful heaters, allowing temperatures typically in the range 4–600 K to be covered.

2.3.5.3.3. *Uniaxial stress*

There are two major applications of *in situ* uniaxial loading. In the first, stress-induced phase transitions, ferroelasticity or simply mechanical response are studied throughout the whole sample as a function of applied stress. This may be undertaken on any powder diffractometer with a reasonable data-collection rate, depending on the resolution required. Parameters typically monitored are the relative phase proportions of parent and daughter structures, lattice parameters, individual peak shifts, which can yield the single-crystal elastic constants (Howard & Kisi, 1999), peak widths, which can indicate the breadth of strain distributions, and preferred-orientation parameters, which can indicate the degree of ferroelasticity (Kisi *et al.*, 1997; Ma *et al.*, 2001; Forrester & Kisi, 2004; Forrester *et al.*, 2005). The second application involves strain scanning using an engineering diffractometer as described in Section 2.3.4.3; however, in this instance an external load is applied to the object under study. This technique can be used to validate finite element analysis simulations of complex components with or without internal residual stresses.

Devices for the *in situ* application of uniaxial stress include adaptations of laboratory universal testing machines such as the 100 kN hydraulic load frame shown in Fig. 2.3.27. Devices such as this may be used in tension, compression, fatigue or even creep conditions depending on the sample and the problem under study.

For more specialized applications, it is sometimes possible to create a more compact device. A recent adaptation of strain



(a)



(b)

**Figure 2.3.28**

(a) Cross section and (b) exterior of a self-loading die for the study of stresses in granular materials.

scanning is to study the stress distribution within granular materials subjected to a variety of load cases as either the average stresses shown in Fig. 2.3.23 (Wensrich *et al.*, 2012; Kisi *et al.*, 2014), or the stress tensor in individual particles throughout a granular material bed. The latter provides insight into inhomogeneous stress distributions such as force chains (Wensrich *et al.*, 2014). The device that was used in these studies (Fig. 2.3.28) is a self-loading die within which a granular material is compacted while diffraction studies are conducted.

## 2.3.6. Concluding remarks

Neutron powder diffraction is just one of many neutron-scattering techniques available; however, it is one that is very commonly used. In fact, the demand for this particular neutron technique is rivalled only by that for small-angle neutron scattering. The close analogy with X-ray powder diffraction makes the technique very familiar to many practitioners of that technique. The differences from X-rays are also critical (Sections 2.3.1 and 2.3.2), since these are the means by which neutron diffraction can obtain information not otherwise accessible. In this chapter we have included descriptions of the various types of neutron source, the neutron powder diffractometers installed at these sources, and a selection of routine and more specialized applications. Demand for the technique is expected to continue, buoyed by further developments in instrumentation and the exploration of new applications.

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